

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Production of Granular Halogen-Containing Polymers

We, CHEMISCHE WERKE MUNCHEN OTTO BARLOCHER G.m.b.H., a German Company, of 16 Reissstrasse, 8 Munich 54, Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The specification of our Patent Application No. 10445/67 (Serial No. 1178847) discloses a process for the production of a granular product from a pulverulent material selected from the group of plastics processing additives consisting of pulverulent inorganic and/or metal soap stabilisers, fillers, dyestuffs, antistatic agents and fire-retarding agents, which comprises mixing the selected pulverulent material or materials with a granulating agent consisting of at least one organic compound which has a melting point above 40°C, is solid and non-adhesive at room temperature, and has a molecular weight of above 150, preferably above 200, heating the mixture to melt the granulating agent while vigorously stirring the mixture, and then cooling the resultant hot mix to room temperature while under reduced stirring to form granules.

Particularly suitable as such granulating agents are those compounds which are usually employed as lubricants in plastics processing. They generally contain a long chain hydrocarbon radical. Especially suitable are spermaceti or other esters derived from fatty alcohols or synthetic alcohols having at least six carbon atoms and fatty acids, esters derived from fatty acids and polyhydric alcohols, and also partial esters such as glycerol monostearate, trimethylol propane distearate or pentaerythritol distearate, fatty alcohols, corresponding synthetic alcohols, fatty acids, corresponding synthetic acids, oxy- or halogen fatty acids.

In addition, paraffin waxes, synthetic paraffin waxes, montax waxes improved montan waxes, hardened vegetable and animal oils such as hardened castor oil for example, ester waxes, fatty acid amides, fatty acid alkylol-amides, fatty acid alkylolamine esters such as triethanolamine distearate, fatty amines, fatty ketones such as stearone, anhydrides of higher carboxylic acids, alkylphenols, and long chain ethers, such as distearyl ether or fatty alcohol polyglycol ether or alkylphenol polyglycol ether for example, are suitable as granulating agents. From 2 to 40%, preferably from 7 to 15%, referred to the pulverulent material to be granulated, may be used.

Granulating agents which are very hard at room temperature and impart a brittle feel to the granules can be softened by means of suitable plasticisers. Hard granulating agents are, for example, the synthetic paraffin waxes and the improved montan waxes having melting points of about 100°C, and ethylene-bis-stearamide having a melting point of about 135°C. However, waxes having lower melting points, such as carnauba wax for example, may also be hard and brittle at room temperature. The usual plasticisers such as mineral oils, chloroparaffins, epoxydised oils such as epoxydised soya bean oil, and also phthalate plasticisers such as dioctyl phthalate, and phosphate plasticisers such as tricresyl phosphate, are suitable for softening purposes.

As a result of research it has been found that the process can be successfully applied for the granulation of halogen-containing polymers prepared by any of the known methods such as those of block polymerisation, suspension polymerisation or emulsion polymerisation. The process is also found suitable for the granulation of vinyl halide copolymers, particularly vinyl chloride/vinyl acetate copolymers, and for the granulation

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of graft polymers and vinylidene chloride polymers.

According to the present invention, therefore, there is provided a process for the production of a granular halogen-containing polymer composition, which comprises mixing a pulverulent halogen-containing polymer with a granulating agent consisting of at least one organic compound having a hydrocarbon radical and having a melting point between 40°C and 150°C and which is solid and non-adhesive at room temperature, heating the mixture to melt the granulating agent while vigorously stirring the mixture, and then cooling the resultant hot mix to room temperature while under reduced stirring to form granules containing pulverulent polymer bound with the granulating agent.

Any of the compounds mentioned as granulating agents in the foregoing and in the aforesaid specification No. 10445/67 are suitable for use as granulating agents in performing the process in accordance with the present invention.

The granulating agent acts in the molten condition at the granulation temperature as a binder for the particles of the pulverulent polymer, and when in the solidified state again at room temperature acts as a parting agent to prevent the formed granules from sticking together. Not only that, but the granulating agent present in the granules performs the function of a parting and mould release agent in subsequent processing of the granular polymer composition. This is an important advantage of the present invention because it is possible and advantageous to utilise as granulating agents those substances which would otherwise subsequently have to be added as lubricants or parting agents in the processing of the polymer. These substances may be selected with melting points in a very wide range and thus may be adapted to the technical requirements of the particular type of subsequent processing for which the granular polymer composition is to be used. For example, esters derived from fatty acids and polyhydric alcohols, such as glycerol monostearate, may be utilised as granulating agents in the production of a granular polyvinyl chloride product, and serving in that product as a compatible lubricant for hard processing of the PVC into transparent or translucent articles of manufacture.

Another advantage of the present invention is that such further processing additives, for example stabilisers, dyestuffs, antistatic agents, fire retarding agents and fillers, as may be required for subsequent processing of the polymer, may also be granulated at the same time during the granulation process and thereby be incorporated in the granules of the granular polymer composition produced. In this way can be avoided the kinds of

errors which are liable to occur in subsequently mixing processing additives with resin powders as has hitherto been the practice in the art, and which may result, for example, from differences in densities; and hence can be avoided the consequential irregularities in the finished plastics article produced. The present invention enables polymer compositions of predetermined and accurately proportioned constituents suited for the various processing purposes, to be manufactured in a simple manner in granular form. The production of a uniform granular product the granules of which contain a desired composition of constituents (halogen-containing polymer and processing additives), which is made possible by this invention, constitutes an important technical advance for the processing, and for the processors, of halogen-containing polymers.

An important technical improvement also lies in the granular form as such, because in contrast to products in fine powder form, the granular form is free-flowing, ensures freedom from dust, and has a virtually indefinite storage life. These qualities permit storage in silos and continuous feeding at controlled rates of flow.

In producing granular halogen-containing polymer compositions by the process according to the invention, the pulverulent halogen-containing polymer is introduced, together with the granulating agent, into a high speed mixed provided with heating and cooling facilities. The mixing mechanism is rotated at more than 1000 rpm for thoroughly mixing the constituents, and the mix is raised to a temperature above the melting point of the granulating agent. In the case of granulating agents having low melting points, for example tallow fatty alcohol having a melting point of about 48°C, this temperature is reached by frictional heat in the high speed mixer, without need for heat to be applied. In the case of granulating agents having higher melting points, for example hardened castor oil which has a melting point of about 84°C, additional heating is applied. After the melting point of the granulating agent has been exceeded, so that the granulating agent is molten, the speed of rotation of the stirring mechanism is reduced to below 700 rpm, and if additional heating was previously applied, its supply is discontinued. The mix is cooled while the slow stirring is continued, until granules form. Depending on the stirring speed, the temperature, and the granulating agent, smaller or larger granules having diameters between 0.1 and 10 mm may be obtained. The preferred granulation temperatures are between 45°C and 55°C.

The following Example illustrates the invention.

## EXAMPLE

- 30 kg of a pulverulent emulsion polyvinyl chloride having a K value of 60 are mixed with 3 kg of cetyl palmitate at 1800 rpm in a mixer of 75 litre capacity, provided with heating and cooling facilities. After a mixing time of about 15 minutes the granulation temperature of about 50°C is reached by frictional heat without additional heating. Stirring is then continued at 600 rpm and cooling is applied. Granules having a mean diameter of 1 mm are obtained.

## WHAT WE CLAIM IS:—

1. Process for the production of a granular halogen-containing polymer composition, which comprises mixing a pulverulent halogen-containing polymer with a granulating agent consisting of at least one organic compound having a hydrocarbon radical and having a melting point between 40°C and 150°C and which is solid and non-adhesive at room temperature, heating the mixture to melt the granulating agent while vigorously stirring the mixture, and then cooling the resultant hot mix to room temperature while under reduced stirring to form granules containing pulverulent polymer bound with the granulating agent.
2. Process as defined in claim 1 wherein the granulating agent is an organic compound used as a lubricant in processing the halogen-containing polymer for the production of articles of manufacture therefrom.
3. Process as defined in claim 1 or 2 wherein the halogen-containing polymer is polyvinyl chloride.
4. Process as defined in claim 1, 2 or 3 wherein the granulating agent is selected from the group consisting of esters derived from fatty alcohols or synthetic alcohols having at least six carbon atoms and fatty acids, esters derived from fatty acids and polyhydric alcohols, partial esters, fatty alcohols, corresponding synthetic alcohols, fatty acids, corresponding synthetic acids, oxy- or halogen fatty acids, paraffin waxes, synthetic paraffin waxes, montan waxes, improved montan waxes, hardened vegetable and animal oils, fatty acid amides, fatty acid alkylolamides, fatty acid alkylolamine esters, fatty amines,

fatty ketones, anhydrides of higher carboxylic acids, alkylphenol polyglycol ethers, alkylphenols, fatty ethers, and fatty alcohol polyglycol ethers.

5. Process as defined in any one of the preceding claims wherein the granulating agent is utilised in a proportion in the range 2—40% by weight of the pulverulent halogen-containing polymer.

6. Process as defined in claim 5 wherein the proportion of granulating agent utilised is in the range 7—15% by weight of the polymer.

7. Process as defined in any one of the preceding claims wherein the granulating agent used is an organic compound which is hard and brittle at room temperature, and a plasticiser therefor is incorporated in the mix to soften said compound in the granules formed.

8. Process as defined in claim 1 wherein the mixture is heated to a granulating temperature in the range 45—55°C.

9. Process as defined in claim 1 substantially as described in the foregoing Example.

10. A granular halogen-containing polymer composition when produced by a process as defined in any one of the preceding claims.

11. A granular composition comprising granules composed of a coherent mass of particles of a pulverulent halogen-containing polymer bound with an organic binder which consists of at least one organic compound having a hydrocarbon radical and having a melting point between 40°C and 150°C and which is solid and non-adhesive at room temperature.

12. A granular composition as defined in claim 11 the granules of which contain polyvinyl chloride together with selected processing additives as used in processing polyvinyl chloride into articles of manufacture.

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